VOLUME 70 OF 77 OF SUBMISSION

STUDY TITLE

ANALYTICAL METHOD FOR THE DETERMINATION OF CGA-169374 IN WHEAT RAW AGRICULTURAL COMMODITIES BY GAS CHROMATOGRAPHY WITH NITROGEN/PHOSPHORUS DETECTION

DATA REQUIREMENT

EPA GUIDELINES NO. 171-4

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PERFORMING LABORATORY

CIBA-GEIGY CORPORATION
GREENSBORO, NC

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VOLUME 1 OF 1 OF STUDY

PAGE 1 OF 86

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Date: June 27, 1991

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GOOD LABORATORY PRACTICE STATEMENT

To the best of my knowledge, the GLP statement found on page 19 of this volume, and signed by the study director, is truthful and accurate.

Signature of Agent of Submitter/Sponsor — Date: 6/26/91

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TABLE OF CONTENTS

TITLE	PAGE NO.
AG-575A, "ANALYTICAL METHOD FOR THE DETERMINATION OF CGA-169374 IN WHEAT RAW AGRICULTURAL COMMODITIES BY GAS CHROMATOGRAPHY WITH NITROGEN/PHOSPHORUS DETECTION	5
APPENDIX 1: VOLUMES ACCOMPANYING THIS SUBMISSION	30
APPENDIX 2: AG-575, "ANALYTICAL METHOD FOR THE DETERMINATION OF CGA-169374 IN WHEAT RAW AGRICULTURAL COMMODITIES BY GAS CHROMATOGRAPHY WITH NITROGEN/PHOSPHORUS DETECTION"	31
APPENDIX 3: RESIDUE TEST REPORT RI-MV-011-90	55
APPENDIX 4: AG-537A, "ANALYTICAL METHOD FOR THE DETERMINATION OF CGA-169374 IN WHEAT RAW AGRICULTURAL COMMODITIES BY GAS CHROMATOGRAPHY"	58
APPENDIX 5: AG-A 10646-01	83

ANALYTICAL METHOD FOR THE DETERMINATION OF CGA-169374 IN WHEAT RAW AGRICULTURAL COMMODITIES BY GAS CHROMATOGRAPHY WITH NITROGEN/PHOSPHORUS DETECTION

METHOD NO. AG-575A
(SUPERSEDES METHOD AG-575)
RESIDUE CHEMISTRY DEPARTMENT
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TABLE OF CONTENTS

		Page No.
I.	INTRODUCTION/SUMMARY	
	A. Scope	4
	B. Principle	4
II.	MATERIALS AND METHODS	
	A. Apparatus	5
	B. Reagents	5
	C. Analytical Procedure	6
	1. Sample Preparation	6
	2. Extraction	7
	3. Partition Clean-up	7
	4. Silica Sep-Pak Clean-up	8
	5. Phenyl Bond-Elut Column Clean-up	9
	6. Charcoal Column Clean-up	9
	D. Instrumentation	10
	1. Description and Operating Conditions	10
	2. Calibration and Standardization	10
	E. Interferences	11
	F. Confirmatory Techniques	12
	G. Time Required	12
	H. Modifications	12
	I. Preparation of Standard CGA-169374 Solutions	12
	J. Determination of Sample Residues	12
III.	RESULTS AND DISCUSSION	14
IV.	CONCLUSIONS	14
V.	CERTIFICATION	15

TABLE OF CONTENTS (Continued)

Page No.

VI. CERTIFICATION OF GOOD LABORATORY PRACTICES 15

VII. LIST OF TABLES AND FIGURES

TABLE I: GAS CHROMATOGRAPHIC CONDITIONS FOR ANALYSIS OF CGA-169374 16

TABLE II: TYPICAL STANDARDIZATION DATA FOR A CGA-169374 STANDARD CURVE 17

TABLE III: SUMMARY OF RECOVERY DATA FOR WHEAT SAMPLES FORTIFIED WITH CGA-169374 18

FIGURE 1: Flow Diagram for Analytical Method AG-575A 19,20

FIGURE 2: Typical Standard Chromatograms of CGA-169374 21

FIGURE 3: Typical Chromatograms for the Analysis of CGA-169374 in Wheat Forage 22

FIGURE 4: Typical Chromatograms for the Analysis of CGA-169374 in Wheat Straw 23

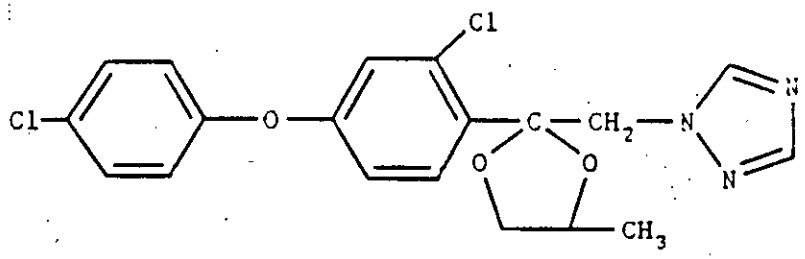
FIGURE 5: Typical Chromatograms for the Analysis of CGA-169374 in Wheat Grain 24

VIII. REFERENCES 25

I. INTRODUCTION/SUMMARY

A. SCOPE

This method is used for the determination of parent residues of CGA-169374 (1-[[2-[2-chloro-4-(4-chloro-phenoxy)phenyl]-4-methyl-1,3-dioxolan-2-yl]methyl]-1H-1,2,4-triazole) in wheat raw agricultural commodities. The limit of sensitivity of the method is 0.10 ng of CGA-169374. The limit of determination for CGA-169374 residues is 0.01 ppm for wheat grain and 0.05 ppm for wheat forage and wheat straw, as demonstrated by fortification experiments. The chemical structure of CGA-169374 is presented below.



AG-575A has been issued to include the following additions and corrections to AG-575[1]:
(1) validation data was added for wheat grain at 0.01 ppm and (2) a typographical error in the REAGENTS section of Method AG-575 was changed from 9:1 ethyl ether:hexane to 9:1 hexane:ethyl ether. AG-575 is a modified version of AG-537A[2]. The major modifications to Method AG-537A are as follows: (1) the aliquot volume was doubled from 20 ml to 40 ml, (2) a phenyl Bond-elut clean-up step (derived from Analytical Method REM 7/86[3]) was incorporated, and (3) the limit of determination was lowered from 0.05 to 0.01 ppm for wheat grain.

B. PRINCIPLE

A representative crop sample is extracted by refluxing in 8:2 (v/v) methanol:conc. ammonium hydroxide. After filtering, an aliquot of the extract is diluted with water and saturated sodium chloride, and partitioned with hexane. The hexane fraction is partitioned with acetonitrile and the acetonitrile fraction is cleaned up on a silica gel Sep-Pak. The sample is then cleaned up on a phenyl Bond-elut, followed by a charcoal:magnesium oxide:celite column clean-up step. The extract is

analyzed by packed column gas chromatography using nitrogen/phosphorus detection.

A flow diagram for the method is presented in Figure 1.

II. MATERIALS AND METHODS

A. APPARATUS

1.0. Chromatography Column, 19-mm i.d., with teflon stopcock.

2.0. Condenser, Allihn, 60-cm.

3.0. Bond-elut, phenyl (Analytichem, #608303-1210-2032).

4.0. Bottle, Boston round, 8-oz.

5.0. Filter paper, Reeve Angel 802 (coarse porosity), and Whatman 2V (medium porosity), 24-cm.

6.0. Flask, Erlenmeyer, 250-ml.

7.0. Flask, round bottom, 50-ml, 100-ml, 250-ml and 500-ml.

8.0. Funnel, filter, 10-cm.

9.0. Funnel, separatory, 250-ml.

10.0. Heating mantle, 500-ml.

11.0. Rotary evaporator, Buchi, or equivalent.

12.0. Scintillation vial, 20-ml.

13.0. Sep-Pak, silica gel (Waters Assoc. #51900).

14.0. Syringe, 20-ml, Luer-Lok.

15.0. Visiprep solid phase extraction manifold (Supelco #57030 or equivalent).

B. REAGENTS

1.0. Acetone, reagent grade (Fisher cat. #A18SK-4).

2.0. Acetonitrile, HPLC grade (Fisher cat. #A998-4).

3.0. Ammonium hydroxide, concentrated (28-30%), reagent grade (Fisher cat. #A669-212).

4.0. Celite 545 (Fisher cat. #C212-500).

5.0. Charcoal, Norit SX-2 (formerly called Norit SG-Extra), acid washed (American Norit, prepared according to the U.S. Food and Drug Administration Pesticide Analytical Manual, Vol. I, PAM I, Section 232.32a).

6.0. 1:2:4 (w/w/w) Charcoal:magnesium oxide:Celite 545 (PAM I, Section 232.32b).

7.0. Ethyl ether, anhydrous, Reagent A.C.S. (Fisher Cat. #E138-1).

8.0. Hexane, HPLC grade (Fisher Cat. #H302-4).

9.0. Magnesium oxide, 98%, A.C.S. Reagent, (Aldrich Cat. #24,338-8, or equivalent).

10.0. Methanol, HPLC grade (Fisher Cat. #A452-4).

11.0. 8:2 (v/v) Methanol:conc. ammonium hydroxide.

12.0. Sodium chloride, reagent grade (Fisher cat. #S271-500).

13.0. Sodium chloride, saturated solution in distilled water.

14.0. Toluene, HPLC grade (Fisher cat. #T290-4).

15.0. 85:15 (v/v) Toluene:acetone.

16.0. 1:1 (v/v) Toluene:acetonitrile.

17.0. 9:1 (v/v) Hexane:ethyl ether.

18.0. Water, distilled.

19.0. CGA-169374 Analytical Standard, CIBA-GEIGY Corporation, P. O. Box 18300, Greensboro, NC, 27419.

C. ANALYTICAL PROCEDURE

1.0. Sample Preparation

Samples are prepared under the general guidelines of the U.S. Food and Drug Administration Pesticide Analytical Manual Volume I, Section 141. Frozen forage and straw are chopped in a Hobart food chopper. Frozen grain is ground in a Wiley Mill.

2.0. Extraction

2.1. Weigh a 20-g subsample from a well-homogenized chopped or ground crop sample into a 500-ml round bottom flask. Add 200 ml of 8:2 methanol:concentrated ammonium hydroxide solution to grain or forage, and 300 ml to straw samples, along with several boiling chips.

2.2. Fit the round bottom flask onto an Allihn condenser, place the flask in a heating mantle and reflux at a rapid boil for two hours.

2.3. Cool the sample to room temperature and filter the sample extract through a Reeve Angel 802 filter paper placed inside a Whatman 2V filter paper into an 8-oz. Boston round bottle.

3.0. Partition Cleanup

3.1. Place a 40-ml aliquot of the filtered extract solution for grain or forage, or a 60-ml aliquot for straw in a 250-ml separatory funnel. Add 100 ml of distilled water and 4 ml of saturated sodium chloride to the separatory funnel.

3.2. Partition the aqueous methanol with 50 ml of hexane by shaking the separatory funnel for 30 seconds. Allow the layers to separate, draw the lower layer into a 250-ml Erlenmeyer flask, leaving any emulsion in the separatory funnel. Add 4 ml of saturated sodium chloride to the separatory funnel to break most of the emulsion remaining. Draw off the aqueous layer and any

remaining emulsion and combine it with the aqueous layer in the 250-ml Erlenmeyer flask.

3.3. Pour the hexane layer from the top of the separatory funnel carefully, so as not to transfer any water droplets, into a second 250-ml separatory funnel.

3.4. Transfer the aqueous fraction back into the first 250-ml separatory funnel and partition with hexane a second time as in Section II.C.3.2.

3.5. Combine the hexane fraction from Section II.C.3.4 with that from Section II.C.3.3 in the second 250-ml separatory funnel. Partition the hexane with two 50-ml portions of acetonitrile, combine the acetonitrile fractions in a 250-ml round bottom flask and evaporate the contents of the flask to dryness on a rotary evaporator at a bath temperature of approximately 40°C.

4.0. Silica Sep-Pak Cleanup

4.1. Dissolve the residue in the round bottom flask in Section II.C.3.5 in 5 ml of toluene.

4.2. Connect a silica Sep-Pak to the Luer fitting on a 20-ml Luer Lok syringe barrel and prewash the Sep-Pak with 5 ml of toluene. Load the toluene solution from Section II.C.4.1 onto the Sep-Pak. Discard the eluate.

4.3. Rinse the 250 ml round bottom flask with 5 ml of toluene and load the toluene onto the silica Sep-Pak. Discard the eluate.

4.4. Elute the compound, CGA-169374, from the Sep-Pak with 15-ml of 85:15 toluene:acetone and collect the eluate in a 50-ml round bottom flask.

4.5. Evaporate the contents of the flask to dryness on a rotary evaporator at a bath temperature of approximately 40°C.

5.0. Phenyl Bond-Elut Cleanup

5.1. Dissolve the residue in the 50 ml round bottom flask from Section II.C.4.5 with 3 ml of hexane.

5.2. Connect the phenyl Bond-elut column to a Vac-elut assembly and prewash with 3 ml of hexane and discard the eluate. Note that all Bond-elut operations are performed under low vacuum suction applied to the Vac-elut assembly.

5.3. Load the hexane solution from Section II.C.5.1 onto the Bond-elut and rinse the 50-ml round bottom flask with 3 ml of hexane and load onto the Bond-elut column. Repeat the rinse and load with an additional 3 ml of hexane. Discard the eluates.

5.4. Wash the Bond-elut column with 3 ml of 9:1 hexane:ethyl ether and discard the eluate. Repeat this wash three more times, discarding the eluate.

5.5. Elute the compound, CGA-169374, from the Bond-elut with 2 ml of methanol into a scintillation vial. Repeat this step three more times, collecting 8 ml of methanol in the scintillation vial.

5.6. Transfer the contents of the scintillation vial to a 50-ml round bottom flask. Rinse the scintillation vial with 3 ml of methanol and add to the 50-ml round bottom flask.

5.7. Evaporate the contents of the flask to dryness on a rotary evaporator at a bath temperature of approximately 40°C.

6.0. Charcoal Column Cleanup

6.1. Dissolve the residue in the 50-ml round bottom flask of Section II.C.5.7 in 5 ml of toluene.

6.2. Set up the chromatographic column according to the U.S. Food and Drug Administration Pesticide Analytical Manual (PAM), Vol. I, Section 232.34. Place a plug of glass wool at the bottom of the column. Add 1 g of Celite 545 to the column, tamp, add 6 g of adsorbent mixture (see PAM I, Section 232.32b), 1:2:4 (w/w/w) charcoal:magnesium oxide:Celite 545. Place a small glass wool plug on top of the adsorbent.

6.3. Prewash the column with 100-ml of 1:1 toluene:acetonitrile. Load the toluene solution from Section II.C.6.1 onto the column. Collect the load solution in a 250-ml round bottom flask.

6.4. Rinse the 50-ml round bottom flask with 10 ml of toluene. Collect the rinse as described in Section II.C.6.3. Repeat with another 10 ml rinse of the 50-ml round bottom flask.

6.5. Elute the column with 120 ml of 1:1 toluene:acetonitrile and collect the eluate in the 250-ml round bottom flask.

6.6. Evaporate the contents of the 250-ml round bottom flask to dryness on a rotary evaporator at a bath temperature of approximately 40°C. For wheat grain samples, dissolve the residue in the flask in 1.0 ml of toluene. For forage and straw samples, dissolve in 2.0 ml of toluene.

D. INSTRUMENTATION

1.0. Description and Operating Conditions

See Table I.

2.0. Calibration and Standardization

2.1. The GC system is calibrated with each analytical run by checking the retention time and detector response relative to previous runs. Retention

times should not vary by more than 5% and detector response should not vary more than 10% between runs.

2.2. The GC system is standardized by injecting aliquots of standard solutions of CGA-169374 in a working range of 0.1-2.5 ng/injection. A linear regression function is generated from the data comparing detector response and ng injected. Typical standardization data are presented in Table II and typical standard chromatograms are shown in Figure 2.

2.3. As with any packed column GC system, the column should be sufficiently primed to give an optimal peak shape by deactivating any active sites on the column. This is accomplished by making several injections of sample matrix extracts from Section II.C.6.6 until a constant peak shape and sensitivity are obtained for CGA-169374.

2.4. It may be necessary to increase the N/P element power beyond the recommended operating range in order to obtain sufficient peak height of the lowest calibration standard. A baseline of ca. 100 pA was used to acquire the chromatograms in Figures 2 through 5, using a Hewlett-Packard 5880A gas chromatograph.

E. INTERFERENCES

1.0. Some interferences have been observed as a result of carryover when large standard injections precede control and reagent blank injections. These interferences are particularly pronounced when other than "packed on-column" injection techniques are used. These problems can be minimized by properly maintaining the GC system (frequently changing the septum and glass wool at the head of the column), using direct "on-column" injection, and injecting samples and standards in a sequence where

samples and standards of like concentration are adjacent.

2.0. Analysis of control samples of wheat forage and straw has shown no significant interferences at a screening level of 0.05 ppm. Analysis of control samples of wheat grain has shown no significant interferences at a screening level of 0.01 ppm. No interferences have been observed in reagent blanks.

F. CONFIRMATORY TECHNIQUES

None.

G. TIME REQUIRED

A skilled analyst can carry out the extraction, cleanup and analysis of a set of 4-6 samples in a 24-hour period including GC analysis.

H. MODIFICATIONS

None.

I. PREPARATION OF STANDARD CGA-169374 SOLUTIONS

1.0. Weigh 100 mg of CGA-169374 analytical standard into a 100-ml volumetric flask and dilute to the mark with acetone.

1.1. Make serial dilutions of the 1 mg/ml standard solution with toluene to give a series of injection standards in a range of 0.02 to 0.5 ng/µl of CGA-169374.

J. DETERMINATION OF SAMPLE RESIDUES

1.0. Inject aliquots of sample extracts from Section II.C.6.6 into the gas chromatograph under the same conditions as for standards. Make appropriate dilutions of the samples (if necessary) with toluene to bring the sample peak heights within the range of the standard curve. Compare the peak heights of the unknown samples to the standard curve or enter the peak height into the least squares program to determine the nanograms of CGA-169374 in the injected aliquot.

Typical chromatograms for control and procedural recovery wheat samples are shown in Figures 3 through 5.

2.0. Calculate the residue results in terms of ppm of CGA-169374 by the following equation:

(1) $$\text{PPM CGA-169374} = \frac{\text{CGA-169374 found (ng)}}{\text{mg sample injected}} \times \frac{100}{R\%},$$

where mg sample injected is calculated as follows:

(2) $$\frac{G}{V} \times V_a \times \frac{V_i}{V_f} \times \left(\frac{V}{(V + (W \times M/100))} \right) = \text{mg inj.}$$

G = milligrams of sample extracted
V = the volume of the extraction solvent (ml)
V_f = total volume of final injection solution (µl)
R% = recovery ratio given by equation (3)
V_i = injection volume (µl)
V_a = aliquot volume (ml)
W = weight of sample extracted (g)
M = moisture content of sample (%)

*Moisture correction is needed for crop samples with a moisture content >20%. (The percent moisture is taken from PAM, Vol. I, Section 202).

3.0. Fortification Experiments

The method is validated for each set of samples analyzed by including an untreated control sample and one or more control samples fortified prior to extraction. Wheat forage and straw samples are typically fortified at 0.05 ppm CGA-169374. Wheat grain samples are typically fortified at 0.01 ppm.

3.1. Add up to 2.0 ml of the appropriate standard solution of CGA-169374 to 20 g of the wheat substrate prior to the addition of the extraction solvent for reflux in Section II.C.2.1. Allow the sample to sit for a few minutes before adding the extraction solvent. Adjust the concentration of the fortification

solution so that no more than 2 mL of solution is added to 20-g of substrate if a higher concentration spike is desired.

3.2. Analyze the samples through the procedures of the method as for treated samples.

3.3. Calculate the ppm of CGA-169374 in the samples using Equation 1 excluding the 100/R% recovery factor. Determine the recovery factor by first subtracting the detector response for CGA-169374, if any, in the control sample from the CGA-169374 response in the recovery sample. Then calculate the recovery factor expressed as a percentage (R%) by the equation:

$$(3) \qquad R\% = \frac{\text{ppm CGA-169374 found}}{\text{ppm CGA-169374 added}} \times 100\%$$

III. RESULTS AND DISCUSSION

To date, this method has been used for the analysis of control and CGA-169374-fortified samples of wheat forage, straw, and grain, as well as for field-incurred residues of CGA-169374 on the same crop fractions. A summary of procedural recoveries from fortification experiments appears in Table III.

This method is an extension of AG-537A[2], in which the extractability of ^{14}C-labelled weathered residues has been addressed. Refer to AG-537A for more extensive fortification and recovery information.

The ruggedness of this method has been demonstrated and documented during ruggedness trials of methods AG-537A[4] and AG-575[5].

IV. CONCLUSION

Analytical Method AG-575A is a valid and accurate method for the determination of parent residues of CGA-169374 in wheat forage, straw, and grain.

V. CERTIFICATION

The reports and experimental results included in this study, Laboratory Project I.D. AG-575A, are certified to be authentic accounts of the experiments.

5-17-91
Date

W. T. Beidler
Research Scientist
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Residue Chemistry Department
919-632-2976

VI. CERTIFICATION OF GOOD LABORATORY PRACTICES

The analytical work cited as Supplemental Fortification Data, Table III AG-575A was performed in accordance with Good Laboratory Practice Standards, 40 CFR Part 160.

5/17/91
Date

J. Ross, Ph.D.
Study Director

CIBA-GEIGY CORPORATION
AGRICULTURAL DIVISION
RESIDUE CHEMISTRY DEPARTMENT
GREENSBORO, NC 27419

Table I: Gas Chromatographic Conditions for Analysis of CGA-169374

Instrument:	Hewlett-Packard 5880A Gas Chromatograph with a 7673A Autosampler and N/P Detector
Carrier Gas:	Helium, Flow Rate 22 ml/min.
Detector Gases:	Hydrogen, 3-4 ml/min. Air, 60 ml/min. NPD Element Power 80-100 pA
Column:	3% OV-17 on 80/100 Mesh Chromosorb W-HP (2 ft. X 2.0 mm I.D.)
Injection:	Packed column injection, Autosampler (5µl)
Injector Temperature:	290°C
Detector Temperature:	300°C
Column Oven Temperature:	245°C
Retention Time:	~5 min.

Table II: Typical Standardization Data for a CGA-169374 Standard Curve

ng Injected	Peak Height (uv)
0.10	128
0.10	102
0.20	198
0.20	195
0.50	576
1.00	1104

Linear regression analysis with VG Multichrom (Version 1.8), linear through zero fit selected.

(Analysis File = 23 AG575-2)

Slope: 1.10573E + 3

Intercept: 0.00000

Coefficient of Determination: 0.99758

Table III: Summary of Recovery Data for Wheat Samples Fortified with CGA-169374

A. Fortification Data*

Substrate	Fortification, ppm	Recovery (%)
Forage	0.0	<0.05 ppm
	0.05	(76%)
Straw	0.0	<0.05 ppm
	0.05	(122%)
	0.075	(85%)
	Avg =	(104%)
Grain	0.0	<0.01 ppm
	0.0	<0.01 ppm
	0.01	(70%)
	0.01	(85%)
	0.01	(83%)
	0.01	(79%)
	Avg =	(79%)

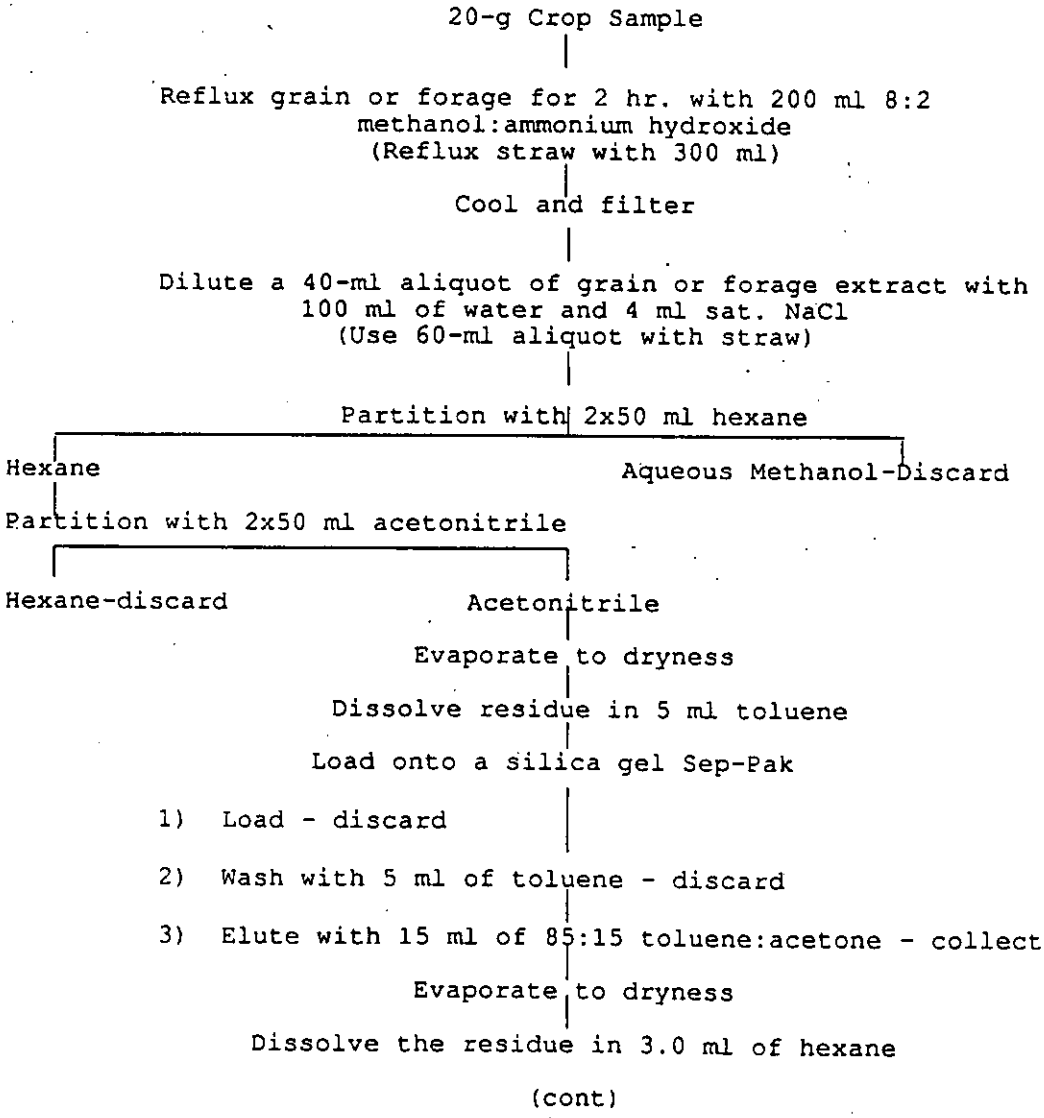
B. Supplemental Fortification Data**

Substrate	Fortification, ppm	Recovery (%)	Field Test #
Grain	0.01	(79%)	MW-FR-703-89
	0.01	(89%)	MW-FR-504-89
	0.01	(67%)	04-FR-001-89
	0.01	(88%)	04-FR-001-89
	0.05	(83%)	MW-FR-603-89
	0.05	(87%)	MW-FR-503-89
	0.05	(70%)	0W-FR-609-89
	0.20	(75%)	04-FR-001-89
	0.10	(109%)	0W-FR-614-89
	1.00	(97%)	0S-FR-103-89
	1.00	(100%)	0S-FR-505-89
	1.00	(78%)	0S-FR-208-89
	0.05	(100%)	05-FR-004-89
	0.05	(102%)	MW-FR-304-89
	0.01	(91%)	0W-FR-504-89
	0.05	(90%)	02-FR-008-89
	Avg =	(88%)	

*Results generated during Method AG-575 validation, RTR No. RI-MV-011-90.

**Supplemental results were generated as part of Study Protocol 19-89 Part-B2 Amendment No. 9, and are reported in ABR-90043 and ABR-90043 Amendment No. 1.

Figure 1: Flow Diagram For Analytical Method AG-575A



(cont)

Figure 1: Flow Diagram For Analytical Method AG-575A

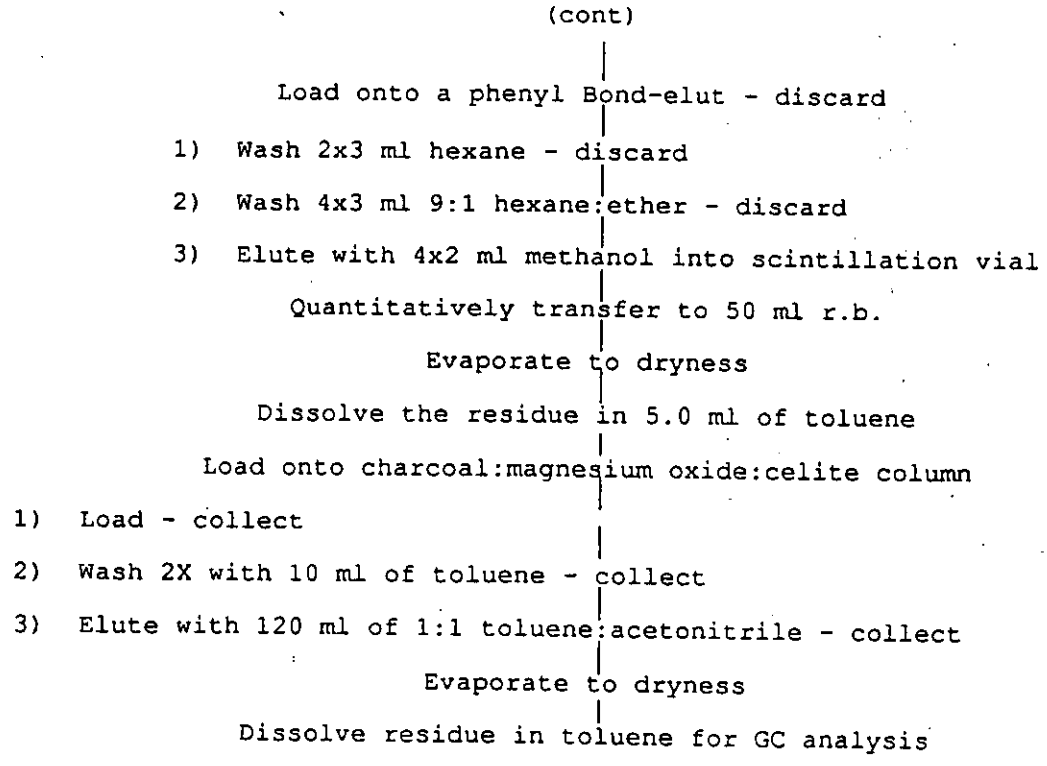
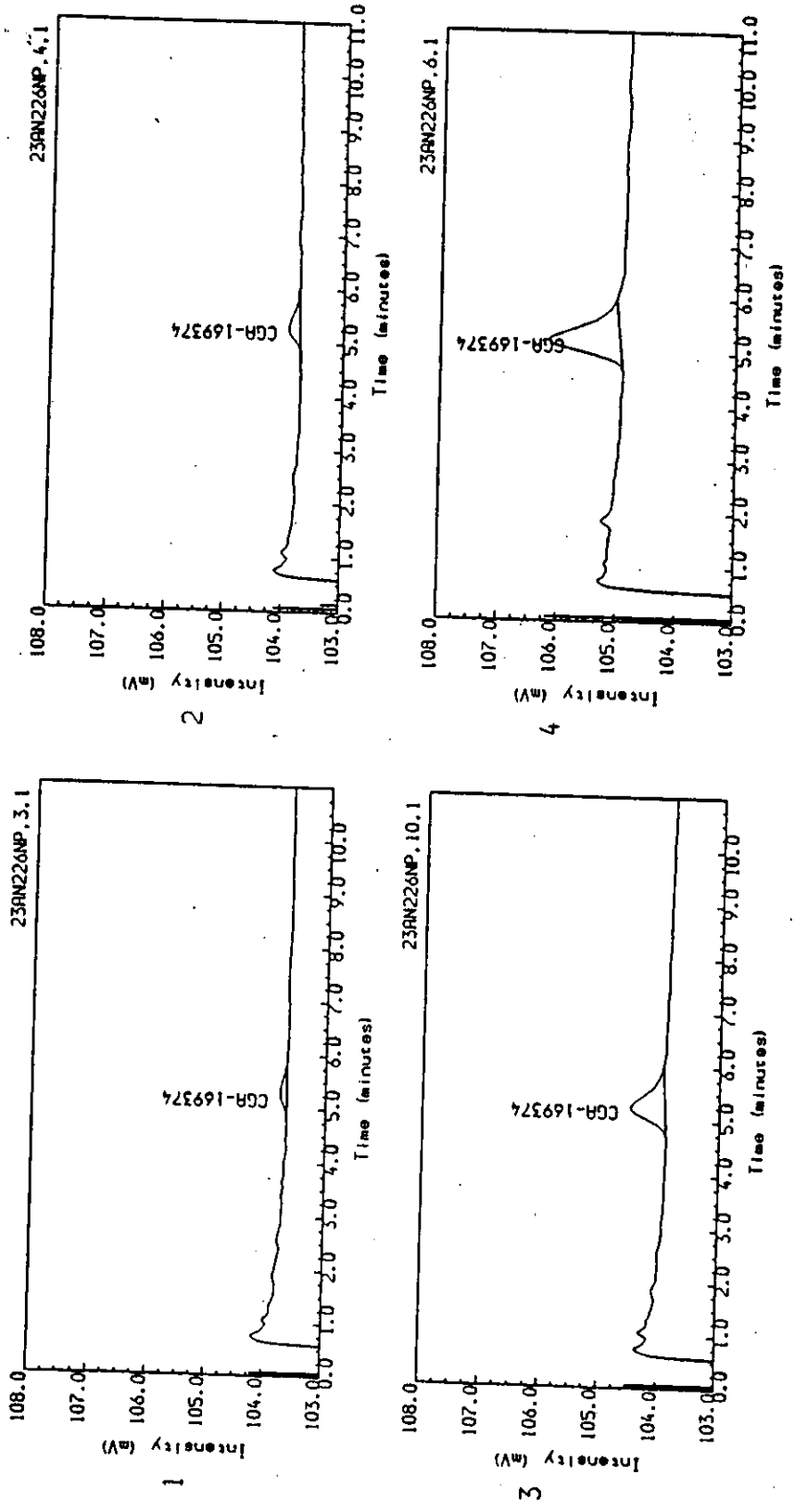
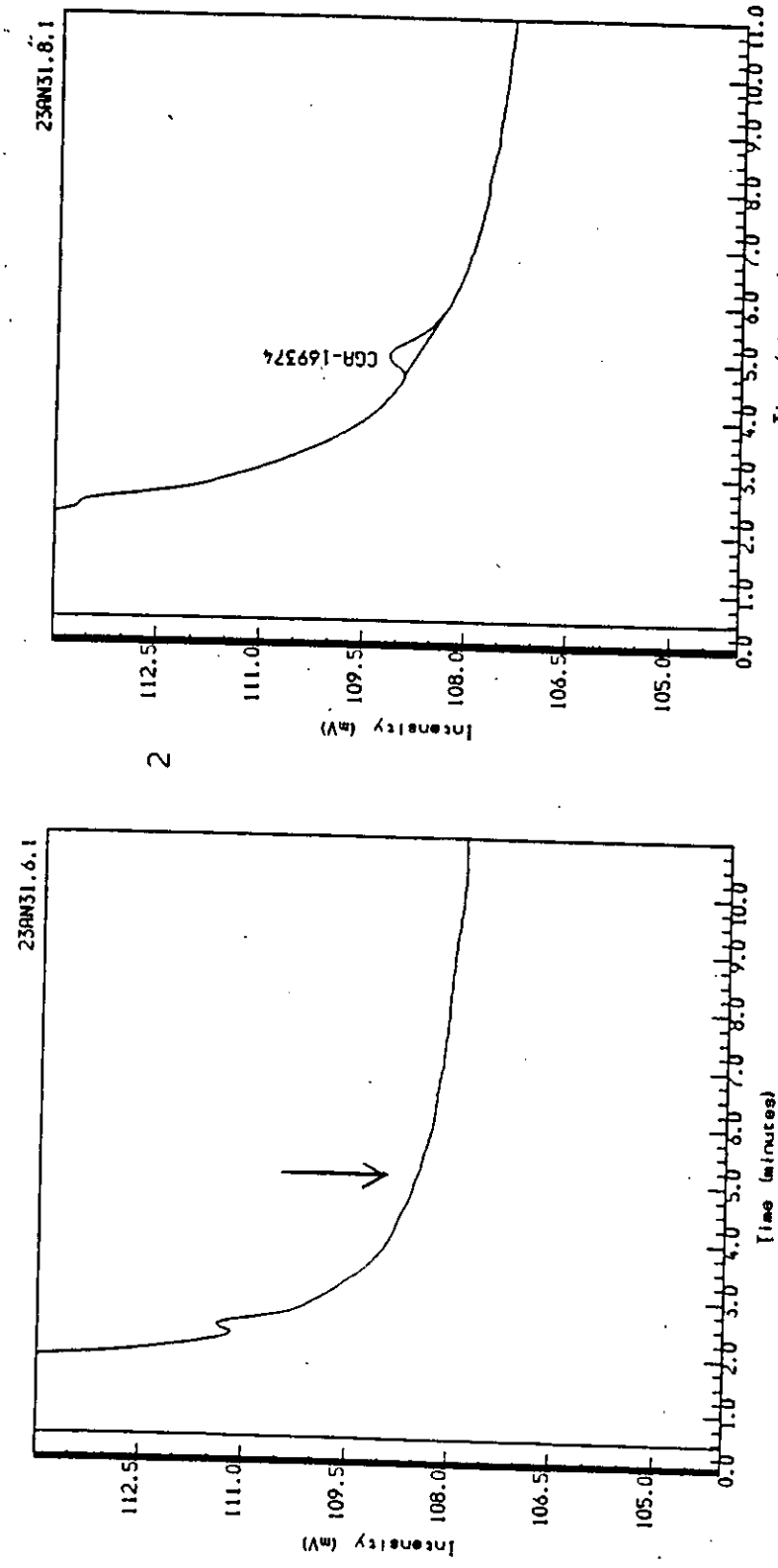


Figure 2. Typical Standard Chromatograms of CGA-169374 Under Analytical Conditions Specified in Table I



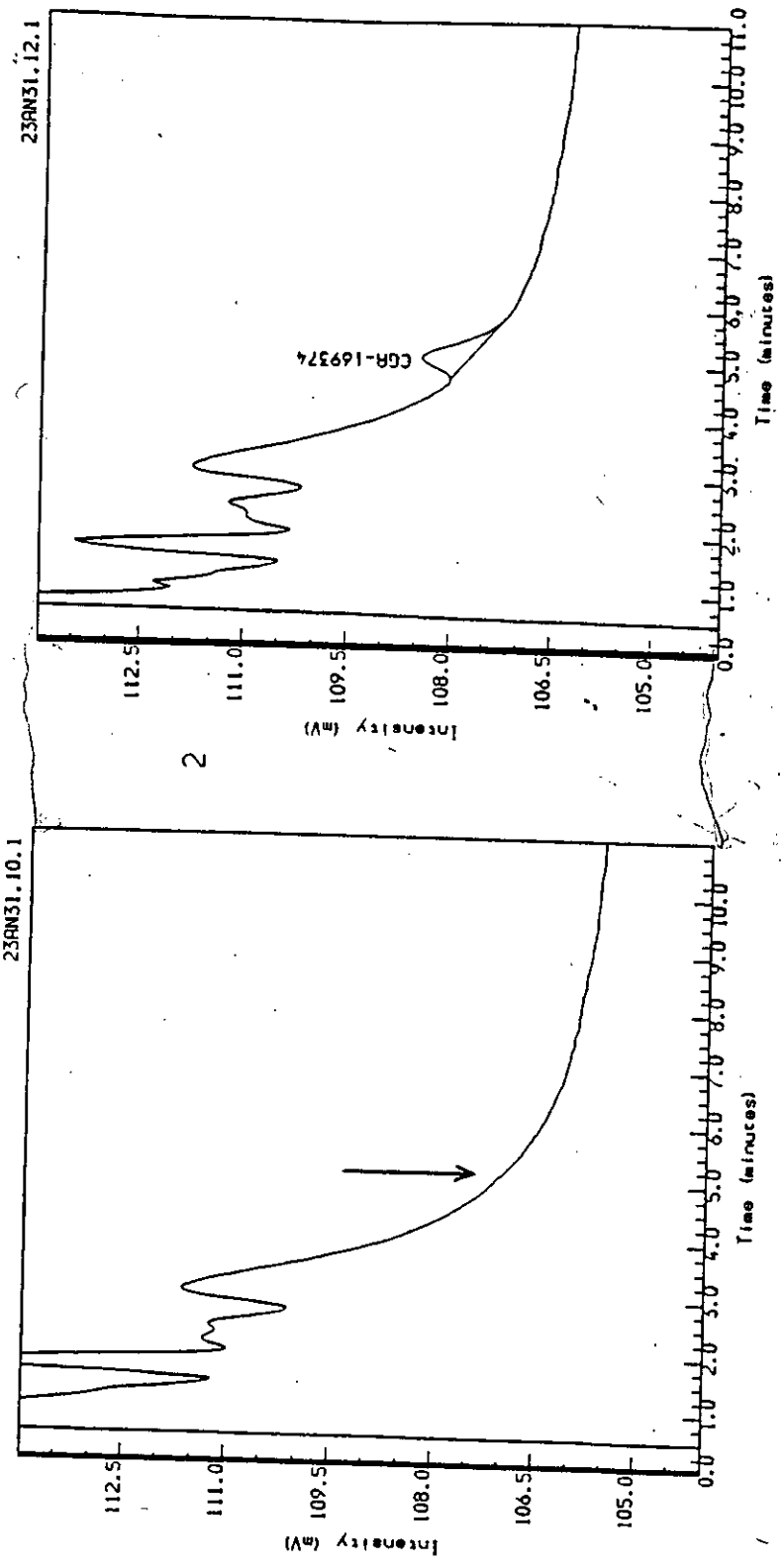
1. 0.10 ng Standard CGA-169374 (Detector response = 109 uV)
2. 0.20 ng Standard CGA-169374 (Detector response = 225 uV)
3. 0.50 ng Standard CGA-169374 (Detector response = 575 uV)
4. 1.00 ng Standard CGA-169374 (Detector response = 1186 uV)

Figure 3. Typical Chromatograms of CGA-169374 in Wheat Forage
Control and Control Fortified with 0.05 ppm CGA-169374



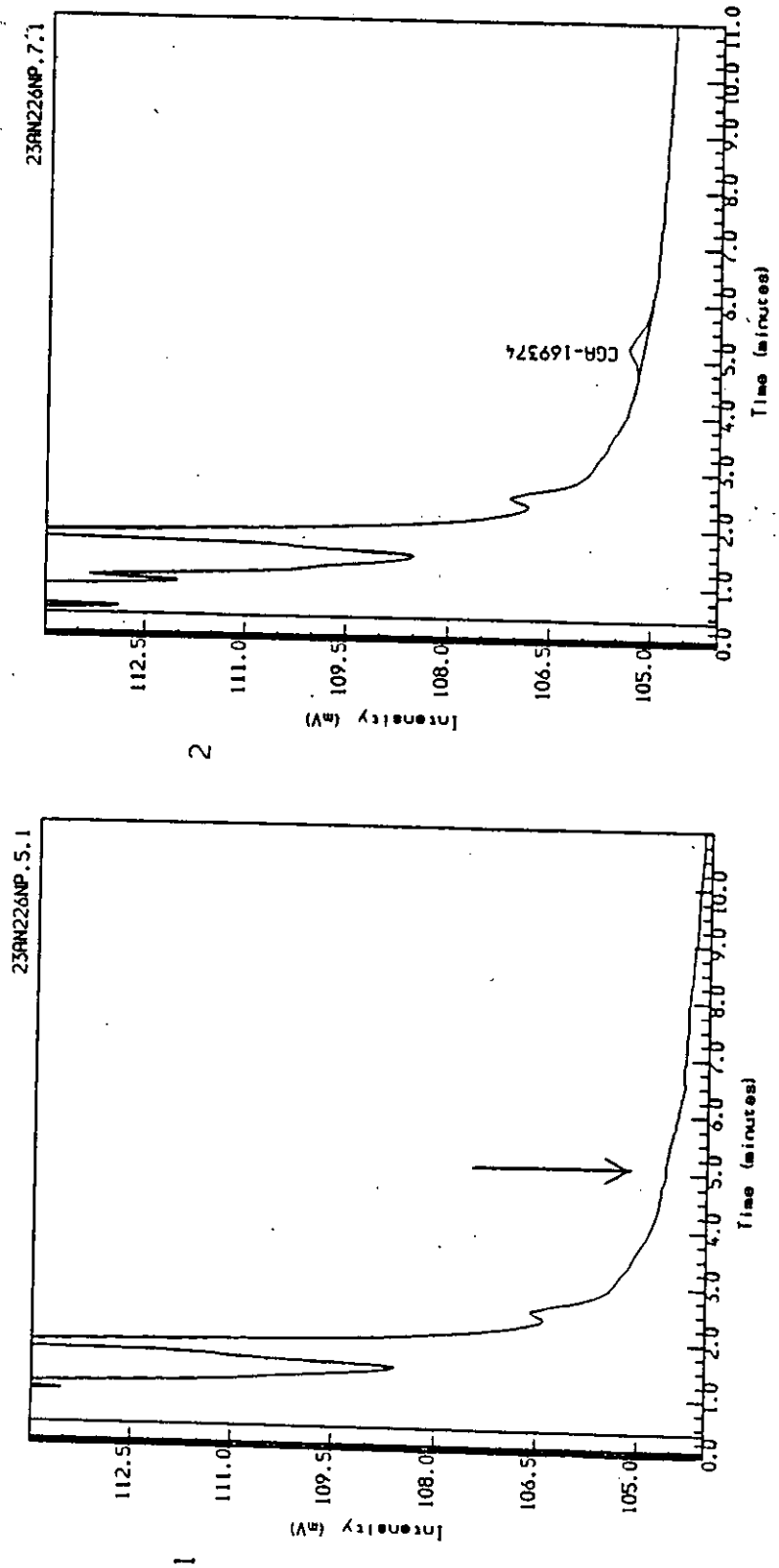
1. Control Forage. 10 mg injected. (0.01 ppm CGA-169374
2. Control Forage + 0.050 ppm CGA-169374, 10 mg injected. 76% Recovery

Figure 4. Typical Chromatograms of CGA-169374 in Wheat Straw Control and Control Fortified with 0.075 ppm CGA-169374



1. Control Straw, 10 mg Injected. (0.01 ppm CGA-169374
2. Control Straw + 0.075 ppm CGA-169374, 10 mg Injected. 85% Recovery

Figure 5. Typical Chromatograms of CGA-169374 in Wheat Grain Control and Control Fortified with 0.01 ppm CGA-169374



1. Control Grain. 20 mg injected. (0.01 ppm CGA-1693744
2. Control Grain + 0.01 ppm CGA-169374. 20 mg injected. 85% Recovery

VIII. REFERENCES

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5. Yarko, J., CYAL Report No. 900201, "Independent Laboratory Confirmation of a Proposed Tolerance Enforcement Method for the Determination of CGA-169374 in Wheat Raw Agricultural Commodities by Gas Chromatography, CIBA-GEIGY Method No. AG-575."

6. Cheung, M. W., Oakes T. L., Moore, M. E., and Smith, J. W., CIBA-GEIGY Residue Dept. ABR-90043, "CGA-169374 (Difenoconazole) - Magnitude of Residues in Spring Wheat and Processed Grain Fractions Following Seed Treatment with Dividend® 3FS."

7. Senzel, A. J. and Ross, J. A., CIBA-GEIGY Residue Dept. ABR-90043, "CGA-169374 (Difenoconazole) - Magnitude of Residues in Spring Wheat and Processed Grain Fractions Following Seed Treatment with Dividend 3FS. Amendment 1 to Incorporate Residue Results for CGA-169374 (Difenoconazole) in Winter Wheat Forage, Straw, and Grain Following Seed Treatment with Dividend 3FS."

APPENDIX 1

VOLUMES ACCOMPANYING THIS SUBMISSION

CIBA-GEIGY LTD. METHOD NO. REM 7/86, "CGA-169374/DETERMINATION OF PARENT COMPOUND BY GAS CHROMATOGRAPHY"

EMS HERITAGE LABORATORIES REPORT NO. EMS9003.1, "METHOD RUGGEDNESS TRIAL FOR CIBA-GEIGY ANALYTICAL METHOD NO. AG-537A FOR THE DETERMINATION OF CGA-169374 IN WHEAT RAW AGRICULTURAL COMMDITIES BY GAS CHROMATOGRAPHY"

CYAL REPORT NO. 900201, "INDEPENDENT LABORATORY CONFIRMATION OF A PROPOSED TOLERANCE ENFORCEMENT METHOD FOR THE DETERMINATION OF CGA-169374 IN WHEAT RAW AGRICULTURAL COMMODITIES BY GAS CHROMATOGRAPHY, CIBA-GEIGY METHOD NO. AG-575"

CIBA-GEIGY RESIDUE DEPARTMENT ABR-90043, "CGA-169374 (DIFENOCONAZOLE) - MAGNITUDE OF RESIDUES IN SPRING WHEAT AND PROCESSED GRAIN FRACTIONS FOLLOWING SEED TREATMENT WITH DIVIDEND 3FS"

CIBA-GEIGY RESIDUE DEPARTMENT ABR-90043, AMENDMENT 1, "CGA-169374 (DIFENOCONAZOLE) - MAGNITUDE OF RESIDUE IN SPRING WHEAT AND PROCESSED GRAIN FRACTIONS FOLLOWING SEED TREATMENT WITH DIVIDEND 3FS. AMENDMENT 1 TO INCORPORATE RESIDUE RESULTS FOR CGA-169374 (DIFENOCONAZOLE) IN WINTER WHEAT FORAGE, STRAW, AND GRAIN FOLLOWING SEED TREATMENT WITH DIVIDEND 3FS"